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ADHESIVE DEVELOPMENT FOR MILITARY BRIDGING
Summary Report

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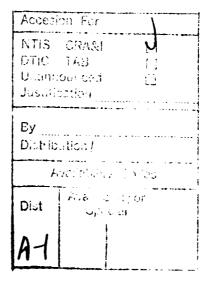
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### ADHESIVE DEVELOPMENT FOR MILITARY BRIDGING

### I INTRODUCTION

The objective of this effort was to develop a family of adhesives, which were specially suited to structural bonding of U.S. Army bridge members both at initial manufacturing and in field repair. The materials to be joined were: aluminum-to-aluminum, aluminum-to-composite, and composite-to-composite. The optimum adhesives should be non-brittle, have high shear strength, high modulus, minimal change in strength and stiffness with relative humidity changes between -65 and 165°F. These structural properties should be retained after prolonged exposure to adverse environments. Processing characteristics were to include: at least one year shelf life, adequate work life, fast cure-to-handling, and insensitivity to minor processing errors.

To accomplish this, base resins with hydrophobic character and curatives with low-temperature reactivity were formulated using structure-property considerations. Phase I took place in the first year of the program and was devoted to base resin development, formulation and screen testing to establish feasibility of tailoring the base resin system from readily available materials. Phase II was slated to occur during the second year and to focus on resin system optimization and further formulation to develop the base adhesive system. Phase III was intended to occur during the remaining nine months and focus on product testing in terms of physical and mechanical properties. This summary report of ingredients, methods, and understanding is the product of this effort.

### II SUMMARY

A coordinated program was undertaken of testing systems comprising suitably hydrophobic epoxy-functional resins and relatively hydrophobic curatives with low-temperature activity

for humidity resistance as well as adhesive toughness. The ultimate basis of humidity-resistance testing was the determination of a quasi-equilibrium stress-strain curve under high humidity at a temperature near the expected upper service temperature (85% RH/120°F). This required extensive stress relaxation at these conditions. At the time that the program was halted, eight experimental formulations had been brought into this test; one of these showed considerable promise of humidity resistance and also demonstrated adhesive toughness. A number of other formulations were sufficiently promising (in preliminary humidity exposure) to be scheduled for this test. Concurrently, methods of toughening were being developed for incorporation into the humidity-resistant systems. These latter methods included use of rubber-based tougheners, flexibilizing components, and fine aluminum particles that are known to have toughening effects.

The most promising system that has been tested is designated BRD-20. It comprises an epoxy resin blend of a multifunctional glycidylated amine (Epon HPT 1071) with the workhorse bisphenol A-based epoxy resin, Epon 828; the curactive mixture includes a toughening rubber-containing polyamidoamine and a low-temperature active modulus-raising diamine, MXDA. Further exploration of this and other routes to humidity resistance combined with adhesive toughness will be necessary.

### III DESIGN OF RESIN SYSTEMS FOR WATER RESISTANT ADHESIVES

As a general rule, water absorption of organic resins is dependent on the overall polarity of the resin. Various rules-of-thumb have been proposed to express or predict the amount of water absorbed (Ref. A). Based on these rules, it is generally desirable to increase the nonpolar (aliphatic or aromatic hydrocarbon) content of the cured resin, and to decrease the content of polar atoms. Resins and curatives containing sizeable

hydrocarbon regions should be explored. Among such resins are the "hydrocarbon epoxy novolacs" (HEN) recently introduced.

Certain specific aspects of room temperature curing agents must also be considered. By far the most effective room temperature curing agents for epoxy resins are primary aliphatic amines. Also, epoxy resins that can be cured at room temperature are almost invariably glycidyl ethers or glycidylamines: products of the reaction of epichlorohydrin with hydroxyl or amine compounds. Curing of glycidyl ether by primary amine results in the following structure (assuming complete reaction):

Note the grouping of five polar atoms (O, N) at intervals of two C atoms. This spacing permits ready H-bonding of water in a five-membered ring, and so facilitates water absorption.

If the curing agent itself also has this two-atom spacing (e.g., DETA, TETA), the structure produced by complete cure may have twenty or more such spacings in close proximity, creating sizeable regions hospitable to absorbed water. One strategy to avoid such regions is to use curing agents (di- and triamines) in which several nonpolar carbon atoms intervene between the amine groups.

Several suitable amine curatives are readily available. If they introduce CH<sub>2</sub> sequences, however, they act as flexibilizing agents, lowering molecular stiffness drastically. To counter this effect, tight molecular branching is necessary. High functionality (number of functional groups per molecule) is one route to such branching. Epoxy novolacs are a group of such polyfunctional resins. Other such are triglycidyl and tetraglycidyl ethers such as Tactix 742 and Araldite MT0163, and polyglycidylamines such as Epon HPT1071.

Primary amine curatives with spacings larger than two carbon atoms include m-xylylenediamine, PACM, and BAC or bis(aminomethyl)cyclohexane, all of which have relatively stiff moieties between the primary amine groups, and dimer diprimary amine, dimer diaminopropylamine, BHMT, hexamethylenediamine, Dytek A, and dodecanediamine, which have aliphatic chains long enough to contribute considerable flexibility.

Since chain flexibility, by lowering glass transition temperature (Tg), also contributes to possible water absorption, it is not possible, a priori, to predict the net effect of changes in resin and curing agent; experimental determination is vital. In a similar way, the effect of water absorption on cured-resin modulus must be experimentally evaluated, as well as the interaction between water absorption and tensile (i.e., dilational as well as shear) modulus. It is for this reason that both weight gain and stepped stress relaxation must be determined in evaluating cured-resin matrix systems.

### IV RESULTS AND DISCUSSION

### A. Resin Blending and Processibility

The materials selected for trial adhesive systems based on these guidelines are listed in Table I. Specific formulations are shown in Table IIA and IIB. Initial systems chosen for investigation were based on high functionality, hydrophobic epoxy resins cured by low temperature-active hydrophobic curatives, accelerated as necessary. Reactive diluent diepoxides were used to modify formulations to permit proper mixing and thin-sheet casting. Successful formulations are listed in Table IIA and those needing further modification are listed in Table IIB. Upon determining moisture resistance of the adhesive systems, those with low water uptake were selected for further modification with toughening agents to improve mechanical properties. In all 37 successful formulations and four commercially available systems were tested. Eleven attempted formulations (Table IIB) were

unsuitable for further investigation, but some served as starting points for more successful formulations.

Specifically, base resins investigated were hydrocarbon epoxy novolacs (HEN XP71756.00 and .01), epoxy phenol novolacs (DEN 438), diglycidyl ether of bisphenol A (Epon 828), bis(diglycidylaminophenylisopropyl)benzene (Epon HPT1071), multifunctional epoxies like tris(p-glycidoxyphenyl)methane (Tactix 742) and tetrakis(p-glycidoxyphenyl)ethane (Araldite MT0163), and experimental fluoroepoxies from Allied. The first five were the most extensively investigated. Reactive diluent resins, resorcinol diglycidyl ether (Heloxy WC69), 1,4-butanediol diglycidyl ether (Heloxy WC67), and triglycidyl ether of glycerol (RF 2500), were used to improve processibility of some systems. Various curing agents, from the well known diethylenetriamine (DETA) and triethylenetetraamine (TETA) to the less common meta-xylylenediamine (MXDA) and bis(paraaminocyclohexyl) methane (PACM), were used. Of the ones investigated, the most useful were MXDA and PACM. Accelerators and modifiers were incorporated in a few formulations, also.

Flexibilizers used were diglycidyl ester of dimer acid (Epon 871), dimer di(aminopropylamine) (DD-3680) and dimer diprimary amine (DF-3680). The systems were toughened with a CTRN-modified polyamidoamine (Ancamide 2033), EC803 (a CTBN adduct of Epon 828 prepared in-house), and atomized aluminum filler (Alcan 201). Note that flexibilizers and tougheners could be obtained with either epoxy resin or amine curative functionality.

### B. First-Level Screening

The first-level screening of the various formulations indicated after a 24 hour exposure what level of moisture resistance was present in the formulated adhesive system. The results are shown in Table III. They are listed in the order of decreasing weight gain. Of the 39 systems tested, about 25 showed a moisture absorption of less than 3% in the preliminary

test. Fourteen systems, including three commercial formulations, showed large moisture absorption (greater than 3%); about four showed inhomogeneity in curing. Five systems were eliminated from further investigation because of these considerations, but several of nearly equal behavior to these were passed into second-level screening, and were eliminated at that stage.

### C. Second-Level Screening

In all, 35 systems were tested in second-level screening for up to 12 weeks. (One system that had been inadvertently omitted from first-level screening was added in.) The results are shown in Table IV. (The order of listing is the same as in Table III.) Comparison of data obtained from second-level screening after an unexpected runaway in temperature with data from first-level screening shows good correlation in Table V, suggesting that the 24-hour exposure was quite comparable to longer-term exposure for screening purposes. The test apparatus is shown in Figure 1. A graphic presentation of moisture absorption of some of the tested systems is shown in Figure 2. Careful examination of Table IV shows some changes in ranking, particularly among the more water-resistant formulations, compared to Table III, but overall the rankings were nearly the same.

On the basis of these rankings, all of the experimental formulations that had gained 4% or more in weight in Table III or Table IV were eliminated from further consideration. (The commercial system, Epiphen 825A, was retained, however.) Also, eliminated were the fluoroepoxy-based formulations, which showed irregular behavior, including poor homogeneity. Twenty-four experimental systems (and three commercial adhesives) survived these eliminations. These were BRD-07A, BRD-08, BRD-10, BRD-11, BRD-12, BRD-19, BRD-20. BRD-23, BRD-24, BRD-25, BRD-26, BRD-31, BRD-34, BRD-35, BRD-37, BRD-38, BRD-40, BRD-41, BRD-42, BRD-43, BRD-44, BRD-45, BRD-46, and BRD-47. Eleven of these incorporated the HEN resin, while five were based on a blend of Epon HPT1071 and Epon 828, one each on Epon HPT1071 and on DEN resin with a

reactive diluent, and eight others incorporated Epon 828. Two of the latter were blends of Epon 828 with HEN resin. When these 24 systems are looked at from the standpoint of curative, the PACM/DP-3680 blend was used 8 times, MXDA/Ancamide was used 3 times, MXDA alone 9 times, MXDA/DD-3680 once, Ancamide alone once, and TAEA or TETA twice. Chart I summarizes all 24 of these combinations.

Chart I

Curative Resin	PACM/ DP3680(f)	MXDA	MXDA/ DD-3680(f)	MXDA/ Ancamide(q)	TAEA(h) or TETA	Ancamide(q)
HEN	BRD-10(d) BRD-24(d) BRD-26(d) BRD-47(a)	BRD-34(C) BRD-43(b) BRD-45(a,C)		BRD-35 <sup>(C)</sup> BRD-37 <sup>(C,e)</sup>		
1071/ 828	BRD-25 BRD-44(a)	BRD-41(b)		BRD-20		BRD-19
.EN/828	BRD-23	BRD-42(b)				
828	BRD-38	BRD-31 BRD-40(b) BRD-46(a)			BRD-11 BRD-12	
1071			BRD-07A			
DEN		BRD-08				

- (a) Alcan 201 as Toughener
- (b) EC803 as Toughener
- (c) Epon 871 as Flexibilizer
- (d) Different reactive diluents
- (e) Used high-functionality HEN and more Epon 871
- (f) Flexibilizer
- (g) Toughener
- (h) Isomeric amine curatives

The use of Alcan 201 did not appear to affect the water absorption of the cured-resin matrix. Comparing the following pairs which differ by the inclusion of Alcan 201 in the second

member, there is little difference in rate or amount of water uptake in Table IV: BRD-10 vs. BRD-47, BRD-31 vs. BRD-46, BRD-34 vs. BRD-45, BRD-25 vs. BRD-44. The more critical question concerns the quasi-equilibrium stress-strain curve (Mecklenburg test), but the program was discontinued before this could be addressed.

### D. Mecklenburg Testing

The Mecklenburg test procedure was begun as soon as the apparatus was ready to use, with some of the earlier materials that had survived the second-level screening. All in all, two of the commercial adhesives and 13 of the 24 experimental formulations were made up for this testing in the time possible. The last three made up (BRD-34, BRD-43, and BRD-45) were ready just at the time the program was discontinued, so the Mecklenburg test procedure (Ref. B, C) was actually applied to 12 materials, of which 10 are listed in Table VII. Tested but omitted materials were BRD-08 and BRD-35. The latter was omitted because its run was discontinued (owing to program discontinuation) when only initial data had been obtained, and approach to quasiequilibrium stress was dubious. Results with BRD-08 (based on the common epoxy-novolac resin DEN 438) gave such low quasiequilibrium stress values that they were regarded with suspicion; time was not available for a replicate run. As noted in Table VII, two other runs were discontinued considerably earlier than the planned 30-day exposure period, but some quasi-equilibrium values were obtained. In several cases, as noted, the quasiequilibrium stress curve had to be extrapolated (never more than 0.1% along the strain axis) to reach the selected final point for tabulation.

A typical Mecklenburg test run is plotted in Fig. 3. The quasi-equilibrium stress-strain curve is shown as the lower envelope of the plotted points, with some smoothing occasionally needed.

The order of listing results in Table VII is the same as the order in Tables III and IV. It is the order of decreasing weight gain (assumed to be water uptake) in the 24-hour first-level screening test (boiling water or exposure to saturated water vapor at the boiling point). As noted, this order, in Table IV, usually resulted in a similar monotonic decrease in weight gain in the 10-week second-level test (exposure to 120F/85% RH). order should reflect the decreasing order of inherent water affinity of the cured resin matrices. From a thermodynamic viewpoint, the acceleration of stress relaxation by the presence of water in the Mecklenburg test must result from this same inherent water affinity. Consequently, as a first approximation, the same order of listing should result in increasing quasiequilibrium stresses at a given strain. It is quite apparent in Table VII that no such regularity can be found. Indeed, in the initial group of results, the material with highest weight gain (Eccobond 91-9) also exhibited the highest quasi-equilibrium stress level, and what regularity there was appeared to reverse the anticipated order.

In the entire group shown in Table VII, a second high-load-bearing material now appears; this is BRD-20, which could be tested for only a few days before the abrupt discontinuance of the program. Unlike the commercial product Eccobond 91-9, this formulation contains no glass fiber, so its performance may well be subject to improvement when such aids are incorporated. It is noteworthy that this formulation also gave moderately high single-lap-shear results (Table VI).

A possible point to note is the peculiarly poor performance of BRD-12 in the Mecklenburg test, in contrast to the nearly identical material BRD-11. The only difference between these formulations is the isomer content of the curative. BRD-11 used the specific isomer TAEA, which contains only primary amine (-NH<sub>2</sub>) groups and a single tertiary N atom at the central branch point of the molecule. In contrast, TETA, used in equal amount

in BRD-12, is a mixture of isomers and homologues (including some TAEA), of which the principal member is the linear trimer, containing two primary and two secondary (-NH-) amine groups. Although TETA should be slightly less reactive (as was noted during cure of the thin samples), this structure would presumably give rise to less regular branching than TAEA, which in turn might affect both water affinity and mechanical properties of the cured matrix. In fact, as Tables III and IV show, water absorption is hardly affected. On the other hand, quasiequilibrium stress at low strains was noticeably altered, including the shape of the lower envelope. A longer period of exposure of BRD-12 would have been advantageous, as well as replication of these observations. (Also slated for attempted replication would have been Eccobond 91-9. This was the first material tested, and the actual procedure used was slightly different from the later tests.)

### E. Lap-Shear Screening

As a crude means of ranking the potential adhesive capabilities of the experimental formulations, standard (ASTM D1002) single-lap shear specimens were made up using FPL-etched aluminum 2024 as substrate. Of the 24 experimental systems that survived second-level screening, 12 were so made up, including 4 of the 8 in Table VII and 4 of the remaining 5 that had been made up for Mecklenburg testing but could not be included in Table VII. (The remaining 5 in the Mecklenburg-test group were untoughened formulations, of which only BRD-23 contained a flexibilizer.)

Results of these lap shear tests are given in Table VI.
Besides the 8 formulations from the Mecklenburg-test group, 4
systems containing a toughener or a flexibilizer were lap-shear
tested. These were BRD-19, BRD-37, BRD-38, and BRD-40.

Although coupon preparation for this screening test was not intended to meet the expressed needs of the Bridge Adhesive-

Construction program, it was expected that qualitative comparison of the results with experimental materials would serve as a guide to improvement of applicable mechanical properties. Of the materials tested and shown in Table VI, toughened formulations, not surprisingly, gave most of the better results. The most outstanding was BRD-40, a variation of a well-known adhesive composition. BRD-40, which is rubber(CTBN)-toughened Epon 828, undoubtedly would require considerable tailoring to produce a useful adhesive. While it is likely that a similar CTBN-828 combination is the base of some of the many commercial products that have already been tested in related programs, the curative MXDA may not have been used in such products. Note also that HEN-based formulations are sprinkled throughout Table VI. order in Table VI is that of increasing lap shear strength. 43 is disappointingly weak, but it is quite likely that the particular CTBN used to make up EC803, which is of optimum solubility parameter for Epon 828, may be a poor choice in the much more hydrophobic HEN. If further Mecklenburg testing were to be done, certainly the four systems mentioned at the end of the last paragraph should be added to the group that had been selected for test but could not be tested.

Table VI contains only one of the aluminum-bearing formulations, but in it, a comparison of BRD-34 vs. BRD-45 suggests that Alcan 201 was of some value in raising lap-shear strength. More extensive testing would be of value.

### V METHODS

### A. First-Level Screening

Formulations prepared successfully and listed in Table IIA were cast between Teflon as thin sheets (0.010") at room temperature. The cure schedule for the test samples was: seven days at room temperature (after mixing and casting) followed by five hours at  $120^{\circ}F$ . Samples for moisture resistance were generally cut out to be 1" by 2 1/2". These samples were

screened at the first level by immersion testing for 24 hours in boiling water and monitored for physical appearance and weight gain. Prior to immersion the specimen was weighed and after immersion weighed again and examined for obvious signs of swelling, softening, or deterioration. Results of this screening are listed in Table III. This method was later modified to comprise exposing a sample in a pressure cooker for 24 hours at 210°F, 99% RH, 1 PSI gauge pressure, to eliminate leaching out of constituents which had been observed using the 24-hour boiling-water immersion method.

### B. Test Apparatus for Second-Level Screening

After these preliminary tests, the adhesive systems were placed in the standard humidity exposure chamber (86% RH/120°F) for the second-level screening. The exposure test chamber was constructed as shown in the detailed sketch (Figure 1). The chamber constant conditions of  $120^{\circ}F \pm 2^{\circ}F$  were maintained by use of an immersion heater controlled by a Honeywell temperature recorder and the air temperature was controlled by a circulation blower and a low wattage electric heater controlled by another Honeywell temperature controller. The air temperature was monitored by a digital thermocouple and recorded by a strip chart recorder tracking at 1/8 inch per hour. Relative humidity of 85%  $\pm$  3% was maintained using a saturated solution of potassium nitrate (KNO3). The solution was 45% by weight KNO3, i.e. 590 g/l (specific gravity - 1.3117).

Second-level screening test results of these systems are shown in Table IV. The specimens for second-level testing were cast as thin (0.010") sheets as described under first-level screening. Five samples of each formulation were suspended in the standard humidity chamber as shown in Figure 1. Measurements of weight to determine percent increase were made initially, at 24 and 48 hours, and at 5 and 7 days. The second-level test effects were then measured at seven-day intervals for 28 days.

Measurements on some systems were taken for up to 12 weeks at 2 week intervals after the first 4 weeks.

### C. Procedure for Mecklenburg Test

The following instructions were written and provided to the University of Southern California for performance of the Mecklenburg tests.

- 1. Measure thickness and width of specimen at several points along its length. When averaging, do not include data taken near the end, where the specimen will be clamped.
  - a. Avoid using specimens that have necked-down regions, or those where the cross-sectional area changes by more than 10% along the useful length.
  - b. When specimen is ready to mount, obtain a dry starting weight.
- 2. Mount specimen in apparatus and adjust dial so force readout is same as before mounting. Measure effective specimen length between clamps. Record dial reading also.
- 3. Insert apparatus into humidity chamber. Monitor and record initial changes in force. If necessary, readjust dial (and record) to keep force near zero.
- 4. Wait 3 to 4 days to reach initial temperature and humidity equilibrium. Monitor each day and adjust dial (record) if necessary to keep force near zero.
- 5. Use dial to stretch specimen about 0.25%, or to a stress level of about 1000 psi (7MPa or 7 x 10<sup>7</sup> dyn/sq cm), whichever comes first.

- Record force readings, including initial stress relaxation.
   Monitor each day or as needed, at constant dial reading.
   Allow 3 4 days to relax.
- 7. Repeat specimen stretching, by net length change of 0.25% of original length or net stress increase of about 700 psi, whichever comes first.
- 8. Continue, alternating steps 6 and 7 for long enough to approach a leveling off of the "equilibrium" stress - perhaps 10 such steps.
- 9. When specimen is demounted, obtain a final weight as rapidly as possible.

The results of the testing are listed in Table VII. An example of intermittent stress-strain relaxation testing is shown in Figure 3.

### VI REFERENCES

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- C. Albrecht, P., Mecklenburg, M. F., Wang, J. K., and Hong, W.-S., "Effect of Environment on Mechanical Properties of Adhesives", Report No. FHWA/RD-87/029, Final Report, February 1987.

## "YABLE I MATERIALS USED IN FORMULATIONS

RESIN (Source)	DESCRIPTION	EčW(a)
ARALDITE MT0163	Tetraphenylolethane tetraglycidyl ether	230
CIDA GE191/ CTBN Adduct EC803 (in-house)	50% rubber (18% AN) in Epon 828	420 (c)
(Dow)	phenol nevolac	178
EPI-REZ SU-8 (Celanese)	Epoxy novolac of high functionality	230
EPON 828 (SHELL) EPON 871 (Shell)	Liquid Deeda resin (n - 0.1) Dialycidyl ester of dimer acid	430
⊣	Bis(diglycidylaminophenylisopropyl)benzene	160
FLUOROEPOXY CO-DGE	Bis(glycidoxyhexafluoroisopropyl)benzene	261 (c)
FLUOROEPOXY C8-DGE	Bis(glycidoxyhexafluoroisopropyl)perfluorooctylbenzene	470 (c)
HEN XP71756.00 (Dow)	Hydrocarbon epoxy novolac ( $f = 3.2$ )	272
1750		237
742 (	Tris(p-glycidoxyphenyl)methane	162
	Resorcinol diglycidyl ether (b)	127.5
HELOXY WC67 (Wilmington) RF 2500 (Resin	<pre>1.4-Butanediol diglycidyl ether (b) Triglycidyl ether of glycerol (b)</pre>	101 (c) 150
Formulators)		
CURATIVE (Source)	DESCRIPTION	AEW (d)
ANCAMIDE 2033 (Pacific	CTBN-modified polyamidoamine	87
ANCAMINE 1637 (Pacific	Mannich base	55
Anchor)		09
DD-3680 (Vomemine-Witch)	bis(nexametnyiene)triamine (e) Dimor di (eminopropylemine)	135
DP-3680 (Kemamine-Witco)		170
	ylenetriami	
MXDA/C-8 Adduct	Fluoroepoxy C8-DGE-Modified MXDA	47 (c)
(in-house) (in-house)	Fluoroepoxy C8-DGE/Epon 828-Modified MXDA	42 (c)
MXDA (Mitsubishi) PACM (Air Products) TAEA (Aldrich) TETA (E. V. Roberts)	Meta-Xylylenediamine Bis(para-aminocyclohexyl)methane Tris(2-aminoethyl)amine	34 (c) 52.5 (c) 24 (c) 24 (c)
	דד דפרוול זכוופרפרדמוודוופ	

Tertiary amine DESCRIPTION ACCELERA FOR (Source) DEAE (Pennwalt)

2,4,6-tris(dimethylaminomethyl)phenol Diethylaminoethanol

Mannich base

CLASS

DESCRIPTION OTHER (Source)

DMP-30 (E. V. Roberts)

Atomized aluminum filler, 20  $\mu$ m particle size Polysulfide modifier Filler 4X Mica (Mineralite) Alcan 201(Alcan) LP-3 (Thiokol)

(a) Epoxy Equivalent Weight, based on vendor's information if not otherwise indicated.

(b) Reactive diluent

(c) Calculated from formula

Amine-hydrogen Equivalent Weight, i.e., weight per active N-H bond, based on vendor's information if not otherwise indicated (g

(e) Extremely impure, variable product

### TABLE IIA

# FORMULATIONS PREPARED SUCCESSFULLY

					H/R
<u>Designation</u>	Component (a)	PBW	No. of	of Equivalents(b)	Ratio(c)
BRD-01	Resins: DEN 438 Heloxy WC69	75 25	Total	0.42 0.20 0.62	
	Curative: BHMT Amine Accelerator: DEAE	10		0.17 N.A.	0.27
BRD-02	Resins: Tactix 742 Heloxy WC67	65	Total	0.40 0.10 0.50	
	Curative: MXDA Accelerator: DMP-30	17		0.50 N.A.	1.00
BRD-05	Resins: DEN 438 RF 2500	80	Total	0.45 0.13 0.58	
	Modifier: LP-3 Curative: DETA Accelerator: DEAE Filler: 4X Mica	12 12 40		N.A. 0.19 N.A. N.A.	0.33
BRD-05U	(Same as BRD-05, but without		filler.)		
BRD-07	Resins: EPON HPT1071 Heloxy WC69	64 13	TO+a1	0.40 0.10	0, [
	Curatives: MXDA DD-3680	13.5 13.5	Total	0.40 0.10 0.50	
BRD-08	Resins: DEN 438 Heloxy WC69	75	Total	0.42	0, [
	Curative: MXDA Accelerator: DMP-30	21 2	1 3 3 4	0.62 N.A.	)  - 
BRD-10	Resins: HEN XP71756.01 RF 2500	64 17	Total	0.27 0.11 0.38	1.0
	Curatives: DP-3680 PACM	15 15	Total	0.09 0.29 0.38	

0.95	0.95		1.03	1.0	1.0	0.97	1.0
0.526 0.500	0.526 0.500	0.45 0.20 0.65	0.67 0.45 0.20 0.65	0.313 0.263 0.576 0.575	0.313 0.263 0.576 0.412 0.161	$\begin{array}{c} 0.211 \\ \underline{0.263} \\ 0.474 \\ 0.35 \\ \underline{0.11} \\ 0.46 \end{array}$	0.35 0.15 0.50 0.38 0.12
		Total	Total	Total	Total Total	Total	Total Total
100	100 12	80 20	16 80 20 16	50 50 50	50 50 14 14	50 50 18.5 18.5	83 15 20 20
Epon 828 TAEA	Epon 828 TETA	DEN 438 Heloxy WC67	TAEA DEN 438 Heloxy WC67 TETA	Epon HPT1071 Epon 828 Ancamide 2033	Epon HPT1071 Epon 828 es: MXDA Ancamide 2033	HEN XP71756.01 Epon 828 :: PACM :: DP-3680	HEN XP71756.01 Heloxy WC67 :: PACM DP-3680
Resin: Curative:	Resin: Curative:	Resins:	Curative: Resins: Curative:	Resins: Curative:	Resins: Ep Ep Curatives:	Resins: H E Curatives:	Resins: H H Curatives:
BRD-11	BRD-12	BRD-13	BRD-14	BRD-19	BRD-20	BRD-23	BRD-24

1.0	1.0	1.0	1.0	1.0	1.0	1.0
0.313 0.263 0.576 0.438 0.135 0.573	0.35 0.15 0.38 0.12	0.132 0.053 0.185 0.186	0.132 0.053 0.185 0.186 N.A.	0.043 0.043 0.043 N.A.	0.26 0.26 0.26	0.26 N.A. 0.077 0.077
Total	Total	Total	Total			
50 50 23	83 19 20 20	25 25 7.8	25 25 7.8	20 2 20 20 20 0.5	50 8.9 50	8.9 1 20 3.6
Resins: Epon HPT1071 Epon 828 Curatives: PACM DP-3680	Resins: HEN XP71756.01 Heloxy WC69 Curatives: PACM DP-3680	Resins: Epon 828 Fluoroepoxy C8-DGE Curative: MXDA/C-8/828 Adduct	Resins: Epon 828 Fluoroepoxy C8-DGE Curative: MXDA/C-8/828 Adduct Accelerator: DMP-30	Resin: Fluoroepoxy C8-DGE Curative: MXDA/C-8 Adduct Resin: Fluoroepoxy C8-DGE Curative: MXDA/C-8 Adduct Accelerator: DMP-30	Resin: Epon 828 Curative: MXDA Resin: Epon 828	Curative: MXDA Accelerator: DMP-30 Resin: Fluoroepoxy CO-DGE Curative: MXDA/C-8 Adduct
BRD-25	BRD-26	BRD-27	BRD-28	BRD-29 BRD-30	BRD-31 BRD-32	BRD-33

	0.98		1.0		1.0	1.0	1.02	0.975	0.99
0.35		0.35			0.235 0.092 0.327	0.526 0.400 0.124 0.524	0.162 0.063 0.095 0.235 0.092 0.327	0.036 0.447 0.483 0.471	0.3125 0.0357 0.1842 0.5324 0.5294
E	TOCAT	E + C - C	Total	E	Total	Total	Total Total	Total	Total
83 17	13	83 17	9 9 5	31 27 42	<b>ω</b> ω	100 21 21	44 115 41 8	15 85 16	50 15 35 18
Resins: HEN XP71756.01 Epon 871	Curative: MXDA	Resins: HEN XP71756.01 Epon 871	Curatives: MXDA Ancamide 2033	Resins: HEN XP71756.00 HEN XP71756.01 Epon 871	Curatives: MXDA Ancamide 2033	Resins: Epon 828 Curatives: PACM DP-3680	Resins: HEN XP71756.00 HEN XP71756.01 Epon 871 Curatives: MXDA Ancamide 2033	Resins: EC803 Epon 828 Curative: MXDA	Resins: Epon HPT1071 EC803 Epon 828 Curative: MXDA
BRD-34		BRD-35		BRD-37		BRD-38	BRD-39	BRD-40	BRD-41

66.0	•	o C	•	٠	C	0		0.98	1.006		o O	00.
0.211 0.036 0.184 0.431	0.4265	0.350	0.382	0.313	0.438 0.135 6.73	N.A.	0.35	N 0.38	0.526 0.529 N.A.	0.33 0.14	0.35 0.11 0.11	N.A.
Total	100	E- 	1001	E	10tal	TOCAT		IOCAI			TOCAL	IOCAI
50 15 35	14.5	83	13	50	23	21	83 17	13 16	100 18 17	79 21	18.5 18.5	19.5
HEN XP71756.01 EC803 Epon 828	MXDA	HEN XP71756.01 EC803	MXDA	Epon HPT1071 Epon 828	PACM DP-3680	Alcan 201	HEN XP71756.01 Epon 871	MXDA Alcan 201	Epon 828 MXDA Alcan 201	HEN XP71756.01 RF2500	PACM DP-3680	Alcan 201
Resins:	Curative:	Resins:	Curative:	Resins:	Curatives:	Filler:	Resins:	<pre>Curative: Filler:</pre>	Resin: Curative: Filler:	Resins:	Curatives:	Filler:
BRD-42		BRD-43		BRD-44			BRD-45		BRD-46	BRD-47		

PBW = Parts by weight

N.A.= Not Applicable

<sup>(</sup>a) See Table 1 for descriptions

<sup>(</sup>b) Epoxy or amine-hydrogen

<sup>(</sup>c) Amine-hydrogen equivalents/epoxy equivalents

T E IIB FORMULATIONS NAMEDING MODIFICATION

	FORM	ULATITONS N	FORMULATIONS NEEDING MODIFICATION	H/P
Designation	Component(a)	PBW	No. of Equivalents(b)	Ratio(c)
BRD-03	Resin HEN XP71756.00 Curatives: DP-3680 PACM	105 15 15	0.39 0.09 0.29 Total 0.38	0.97
	Diluents: Heloxy WC67 Heloxy WC69 RF 2500	Dilutions attempted dilutions upon cool	Dilutions of 10%, 20%, and 30% we attempted with each of these. Aldilutions reverted to a vitreous upon cooling to room temperature.	were All is solid e.
BRD-04	Resins: Epi-Rez SU-8 RF 2500	42	0.18 0.08 0.08	66.0
	Curatives: DP-3680 Ancamine 1637	10		•
		Formulation	gelled within 20	minutes.
BRD-06	Resins: Araldite MT0163 Heloxy WC67	92 10	0.40 0.10 0.10	C
	Curatives: MXDA DP-3680	13.5		) • •
		Resin di mixable	dilution insufficient to	produce
BRD-09	Resins: Epi-Rez SU-8 RF 2500	42 11.7	0.18 0.08 0.08	
	Curatives: DP-3680 PACM Ancamine 1637	10 9 1		0.95
		Not mixable	ble (marginal).	

BRD-15	Resins: Tactix 742 Heloxy WC67	80 20	0.49	
	Curative: TAEA	15	ις	06.0
		Exothermed at 30	min., could not be	cast.
BRD-16	Resins: Tactix 742 Heloxy WC67	80 20	0.49	
	Curative: TETA	15	ıo	06.0
		Exothermed at 30	min., could not be	ćast.
BRD-17	Resins: Epon HPT 1071 Epon 828	50 50	0.313 0.263	
	Curative: MXDA	19.5		0.
		Casting too brit	too brittle to handle.	
BRD-18	Resins: Epon HPT 1071 Epon 828	50 50	0.313 0.263 0.576	
	Curative: PACM	30		0.99
		Casting too brittle	tle to handle.	
BRD-21	Resins: HEN XP71756.01 HEN XP71756.00 RF 2500	33 38 28.5	0.14 0.19 0.19	
	Curative: MXDA	16	1	0.
		Exothermed, could	ld not be cast.	
BRD-22	Resins: HEN XP71756.01 HEN XP71756.00 RF 2500	52 19 28.5	0.22 0.07 0.19	
	Curatives: PACM DP-3680	18.5 18.5 Total		96.0
		Exothermed, could	ld not be cast.	

0.221	0.314	0.086 0.307
	Total	Total
60 40	7.5	7.5
ns: HEN XP71756.00 Epon 871		Ancamide 2033
BRD-36 Resins:	Cura	

Not a mixable resin blend.

0.98

PBW = Parts by weight
N.A.= Not Applicable
(a) See Table I for descriptions
(b) Epoxy or amine-hydrogen
(c) Amine-hydrogen equivalents

### TABLE III

FIRST-LEVEL SCREENING RESULTS

Except where noted, cured at room temperature/7 days + 120°F/5 hrs.

(-)	Cure to		r Weight G	
Designation (a)	Handling (C)	Boiling Water	210 <sup>O</sup> F <u>Steam</u>	Comments
BRD-01	11 hr	13.6 <sup>(g)</sup>	8.1 <sup>(h)</sup>	Swelled, Softened, Yellowed
BRD-05U	16 hr	11.25		16110#ed
BRD-05	16 hr	11.0		
Epiphen 825AU(b,d)	16 hr	9.75		
Epiphen 825A(b)	16 hr	<sub>9.5</sub> (g)	5.2 <sup>(h)</sup>	Swelled, Softened Yellowed
BRD-39			7.6	Moderate warpage; poor replication
BRD-13	50 min		4.5	No warpage
BRD-14	90 min		4.4	No warpage
BRD-02A(j)	2 hr		4.4	No warpage
BRD-02	2 hr	5.7 <sup>(g)</sup>	4.1 <sup>(h)</sup>	Soft when hot
Hysol 9396 <sup>(n)</sup>			4.1	Poor replication
BRD-08	2 hr	3.7		
BRD-19			3.3	
BRD-07	8-12 hr	3.15		Slight inhomogeneity; uncured pockets of resin
Eccobond 91-9(m)			2.7	
BRD-12	180 min	•	2.7	No warpage
BRD-11	140 min		2.6	Minimal warpage
BRD-35			2.6	Severe warpage
BRD-07A(k)	8-12 hr		2.5	Minimal warpage
BRD-20			2.5	
BRD-41			2.4	Slight to moderate warpage
BRD-37			2.35	Severe warpage
BRD-40			2.15	Slight to moderate

BRD-44			2.05	Slight to moderate warpage
BRD-46			2.0	Slight to moderate
				warpage
BRD-47			2.0	Slight to moderate warpage
BRD-10	8-10 hr		1.75	Moderate warpage
BRD-42			1.6	Slight to moderate
<sub>F-5</sub> (e)		3.2 (f)		warpage Inhomogeneous before
r-3. ,		0.8		cure; cured clear
BRD-38			1.47	Slight warpage
BRD-25	· 		1.44	Slight warpage
BRD-26			1.37	
BRD-24			1.35	
BRD-34			1.32	Moderate warpage
BRD-43			1.25	Slight to moderate warpage
BRD-45			1.18	Slight to moderate
BRD-28	4-5 hr		1.6 <sup>(f)</sup>	warpage Inhomogeneous before
	4 3 111		0.6	& after cure; moderate
				warpage
3RD-23			1.00	Slight warpage
BRD-27	4-5 hr		0.1(f)	Inhomogeneous before
	7 J III		-0.3	& after cure
			0.5	" ATCET CATE

- (a) See Table II for formulations
- (b) Monomer Polymer Dajac, included as a reference system
- (c) At room temperature
- (d) Unfilled version
- (e) Fluoroepoxy C8-DGE/MXDA at H/R = 1.0
- (f) Two ostensibly identical specimens
- (g) Cured at room temperature >5 weeks at time of test
- (h) Cured at room temperature >14 weeks at time of test
- (j) Replicate formulation using fresher Mitsubishi MXDA
- (k) Replicate formulation, more throughly mixed
- (m) Emerson & Cuming, included as a reference system
- (n) Dexter Hysol, included as a reference system

TABLE IV

<u>SECOND-LEVEL SCREENING RESULTS</u>

Weight gain, %(a), at 120°F/85% RH

Designation	n (c) 24 hrs	48 hrs	5 dy	7 dy	2 wk	3 wk	4 wk	6wk	8wk	10 wk	12 wk
BRD-01(b)	5.6	5.7	6.1	6.2 <sup>(d)</sup>	4.8	4.9	5.1	5.1	5.0	5.0	
BRD-05U	4.5	4.8	5.1	5.1	5.2	5.2	5.3	5.4	5.4		
BRD-05	3.8	4.0	4.0	3.9	3.9	3.8	3.8	3.9	3.9		
Epiphen 825AU(C)	4.0	4.2	4.6	4.6	4.9	4.8	5.0	4.9	4.9		
Epiphen 825A(c)	3.6	3.7	3.9	4.0	3.9	4.0	4.1	4.1	4.0	3.9	
BRD-02	2.6	2.9	3.4	3.5	3.5	3.7	3.9	3.9	4.0	4.0	
Hysol 9396	(c) <sub>3.3</sub>	3.8	4.2	4.4	4.6	4.9	5.1	5.2	5.4	(g)	(g)
BRD-08 (e)	2.2	2.3 1.9	2.5 2.0(f	2.6	2.7	2.7 2.1	2.8 2.1	2.8	2.8	2.7	2.6
BRD-19 (e)	1.4	1.8	1.9 1.7(f	1.9	2.1 1.9	2.2	2.3	2.5	2.6	3.0	3.0
BRD-07	1.6	1.7	1.9	2.0	2.1	2.3	2.4	2.6	2.6		
Eccobond 91-9 (c) (b,e)	1.3	1.6	1.6 1.4(f	1.7	1.9 1.5	1.9 1.6	1.6	1.6	1.7	1.9	1.8
BRD-12 (e)	1.2	1.4	1.5 1.6	1.5	1.6	1.7 1.9	1.8	2.1	2.2	2.4	2.3
BRD-11	1.3	1.4	1.5	1.5	1.6	1.7	1.8				
BRD-35	0.5	0.5	0.6	0.6	0.6	0.7	0.8	0.8	0.8	0.8	0.9
BRD-07A(b,	<sup>2)</sup> 1.0	1.2	1.3 <sup>(f</sup>	1.4	1.5	1.7	1.8	2.1	2.4	2.7	2.7
BRD-20 (b,e)	1.1	1.3	1.3 1.2(f	1.5	1.6 1.4	1.7 1.6	1.7	1.9	2.1	2.5	2.3
BRD-41	1.2	1.4		1.6	1.7		1.9		2.1		
BRD-31 (e)	1.1	1.2	1.2 1.1	1.2	1.2	1.2	1.3	1.4	1.5	1.6	1.5
BRD-37	0.5	0.5	0.6	0.6	0.6	0.8	0.9	1.0	1.0	1.1	1.1
BRD-40	1.1	1.2		1.3	1.4		1.4		1.5		
BRD-44	0.5	0.6		0.6	0.7		0.9		1.0		
BRD-46	1.0	1.2		1.3	1.3		1.4		1.4		
BRD-47	0.8	0.8		0.9	0.9		1.0		1.0		

### TABLE IV (Continued) SECOND-LEVEL SCREENING RESULTS Weight gain, %(a), at 120°F/85% RH

Designation	(c) 24 hrs	48 hrs	5 dy	7 dy	2 wk	3 wk	4 wk	6wk	8wk	10 wk	12 wk
BRD-10 (b,e)	0.7	0.9	0.8	0.8	0.7	0.7	0.8	1.3	1.4	1.5	1.3
BRD-42	0.7	0.8		0.9	1.0		1.1		1.3		
BRD-38	0.6	0.6	0.7	0.7	0.7	0.8	0.8	0.9	0.9	0.9	0.9
BRD-25	0.7	0.7	0.7	0.7	0.8	0.9	1.0	1.1	1.1	1.2	1.2
BRD-26 (b-3,e)	0.6	0.8	0.8	0.9	0.9 1.0	0.9	1.0	0.9	0.9	1.0	1.1
BRD-24	0.7	0.8	0.9	0.9	0.9	0.9					
BRD-34	0.5	0.5	0.6	0.6	0.6	0.6	0.7	0.7	0.6	0.7	0.7
BRD-43	0.4	0.5		0.6	0.6		0.7		0.8		
BRD-45	0.5	0.5		0.6	0.6		0.7		0.7		
BRD-28	0.9	0.7	0.2	g) <sub>0.1</sub> (g	<sup>(1)</sup> (-0.2)	(g)(-0.3)	(g)(-o.	3)			
BRD-23	0.5	0.5	0.5	0.5	0.5	0.6	0.7	0.7	0.7	0.7	0.7
BRD-27	0.7	0.4	(-0.3)	(-0.6)	(-1.0)	(-1.3)	(-1.	3)			

Average of 5 specimens except where noted. (a)

<sup>(</sup>b) Average of 4 specimens.(b-3) Average of 3 specimens.

<sup>(</sup>c) See Table III for notes.

Data beyond this time on this sample were affected by artefact: temperature excursion of humidity chamber. Replicate exposure of additional specimens. (d)

<sup>(</sup>e)

<sup>(</sup>f) At 4 days.

<sup>(</sup>g) Erratic weight losses.

TABLE V

COMPARISON OF FIRST-LEVEL SCREENING RESULTS TO R. H. CHAMBER RUNAWAY Except where noted, cured at room temperature/7 days + 120°F/5 hrs.

	After R. H	24-hr Weight Gain, %						
<u>vesignation</u> (a)	Chamber <sup>(C)</sup> Runaway	Boiling Water	210 <sup>0</sup> F Steam	Comments				
BRD-01	4.8(g)	13.6 <sup>(g)</sup>	8.1 <sup>(h)</sup>	Swelled, Softened, Yellowed				
BRD-05U	5.45	11.25		Tellowed				
BRD-05	4.30	11.0						
Epiphen 825AU <sup>(b</sup> ,	d) 5.59	9.75						
Epiphen 825A <sup>(b)</sup>	3.9 <sup>(g)</sup>	<sub>9.5</sub> (g)	5.2 <sup>(h)</sup>	Swelled, Softened Yellowed				
BRD-02	3.5 <sup>(g)</sup>	5.7 <sup>(g)</sup>	4.1 <sup>(h)</sup>	Soft when hot				
BRD-08	3.60	3.7						
BRD-19	3.07		3.3					
BRD-07	3.37	3.15		Slight inhomogeneity; uncured pockets of resin				
Eccobond 91-9(m)	2.65		2.7	or resin				
)-12	2.64		2.7	No warpage				
BRD-11	2.61		2.6	Minimal warpage				
BRD-20	2.39		2.5					
BRD-10	1.46		1.75	Moderate warpage				
BRD-26	1.23		1.37					
BRD-24	1.27		1.35					
BRD-28	0.06	•	1.6 <sup>(f)</sup> 0.6	Inhomogeneous before & after cure; moderate warpage				
BRD-27	(-1.53)		0.1 <sup>(f)</sup> -0.3	Inhomogeneous before & after cure				

<sup>(</sup>a) See Table II for formulations

<sup>(</sup>b) Monomer Polymer Dajac, included as a reference system

<sup>(</sup>c) Runaway of temperature and R.H. for 10 hrs. or more.

<sup>(</sup>d) Unfilled version

<sup>(</sup>f) Two ostensibly identical specimens(q) Cured at room temperature >5 weeks at time test began Cured at room temperature >14 weeks at time of test

<sup>(...)</sup> Emerson & Cuming, included as a reference system

TABLE VI

### LAP SHEAR SCREENING

Designation	Base Resin	Psi (b)	Flexibilizer/Toughener	Est'd Content
<b>⊅−4</b> 3	<sub>HEN</sub> (e)	630	/EC803	/15.0% <sup>(c)</sup>
BRD-10	<sub>HEN</sub> (e,h)	690	DP-3680/	13.5%/
BRD-07	1071 <sup>(h)</sup>	760	DD-3680/	13.0%/
BRD-34	<sub>HEN</sub> (e)	1510	Epon 871/	15.0%/
BRD-25	828/1071 (g)	1530	DP-3680/	15.8%/
BRD-35	<sub>HEN</sub> (e)	1610	Epon 871/Ancamide 2033	14.3%/8.0% <sup>(a)</sup>
BRD-19	828/1071 (g)	1690	/Ancamide 2033	/33.3% <sup>(a)</sup>
BRD-45	<sub>HEN</sub> (e)	1830	Epon 871/Alcan 201	14.1%/5.3% <sup>(d)</sup>
BRD-38	828	1850	DP-3680/	14.8%/
BRD-20	828/1071 (g)	2130	/Ancamide 2033	/10.9% <sup>(a)</sup>
BRD-37	HEN (f)	2140	Epon 871/Ancamide 2033	36.2%/6.9% <sup>(a)</sup>
BRD-40	828	3330	/EC803	/12.9% <sup>(C)</sup>

Contains unknown fraction of CTBN.

<sup>(</sup>b) Single lap shear, ASTM D1002, substrate: FPL-etched aluminum 2024.

<sup>50%</sup> CTBN. (c)

Volume content. (d)

HEN XP71756.01. (e)

<sup>(</sup>f) About 50/50 HEN XP71756.00/HEN XP71756.01

<sup>(</sup>g) 50/50 Epon 828/Epon HPT1071.(h) Reactive epoxy diluent also present.

TABLE VII

### <u>Ouasi-Equilibrium Stress(b)</u> <u>at Selected Strains</u> Cured at Room Temperature/7 days + 120°F/5 Hours

	At Strain=										
Designation (a)	0.25%	0.5%	1.0%	1.5%	2.0%	2.5%	<u>3%</u>				
Epiphen 825A <sup>(a)</sup>	0.70 (100)	1.50 (220)	2.50 (360)	3.75 (545)	4.25 <sup>(g)</sup> (615)						
Eccobond 91-9(a)	2.49 (360)	5.64 (820)	(c)								
BRD-12	0.50 (70)	1.00 (145)	3.50 <sup>(g)</sup> (510)	(d)							
BRD-11	1.58 (230)	2.95 (430)	6.12 (890)	8.63 (1250)	9.26 (1340)						
BRD-07A(a)	1.42 (205)	2.83 (410)	5.20 (755)	7.73 (1120)	10.04 (1455)	10.88 (1575)					
BRD-20	2.70 (390)	5.4 <sup>(g)</sup> (780)	(d)								
BRD-31	1.67 (240)	3.43 (500)	6.85 (995)	9.90 (1435)	12.50 (1810)	15.50 <sup>(g)</sup> (2250)					
BRD-10	0.75 (110)	1.75 (255)	4.25 (615)	6.50 (940)	8.25 (1200)	10.50 (1520)	12.00 <sup>(</sup> (1740)				
BRD-25	1.50 (220)	2.75 (400)	5.25 (760)	7.00 (1015)	8.00 (1160)	10.50 (1520)	(e)				
ькD-23	0.83 (120)	1.66 (240)	5.00 (725)	8.10 (1175)	10.00 (1450)	11.50 <sup>(g)</sup> (1670)					

- (a) See Table III for notes.
- (b) Stress values in MPa (psi), estimated from quasi-equilibrium curve obtained after stress relaxation at 120°F, 85% RH (Mecklenburg procedure).
- (c) Specimen broke at 0.65%.
- (d) Run discontinued early.
- (e) Specimen broke at 2.6%.
- (f) Specimen broke at 3.0%.
- (g) Extrapolated slightly (less than 0.1%).

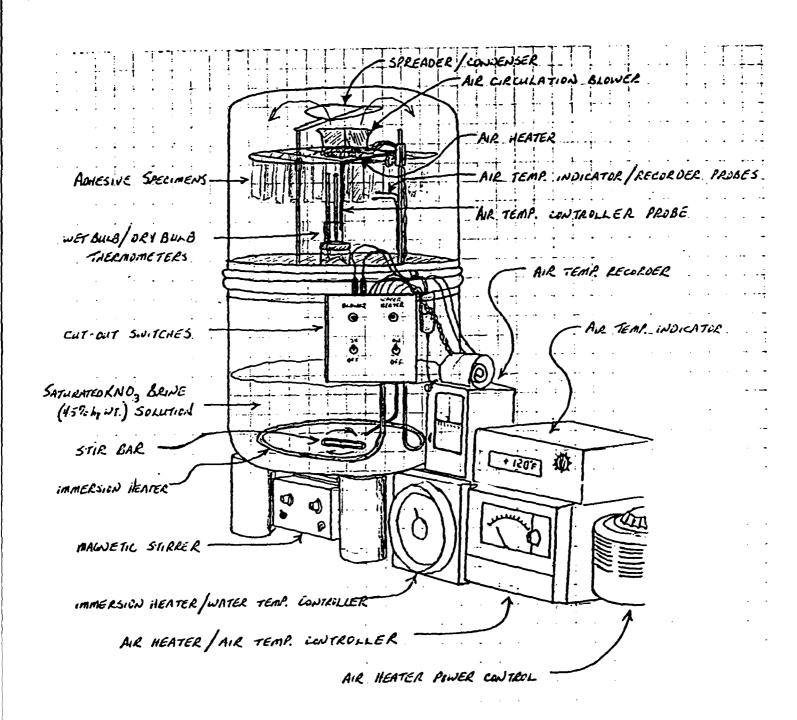


FIGURE 1

SKETCH OF CHAMBER FOR SECOND-LEVEL TESTING

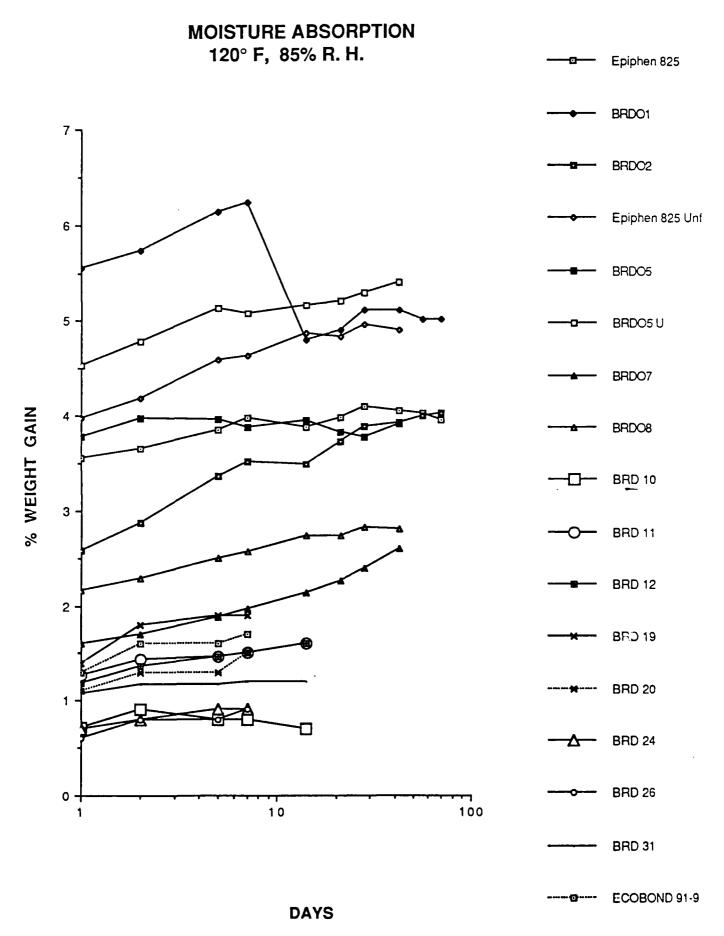


FIGURE 2.

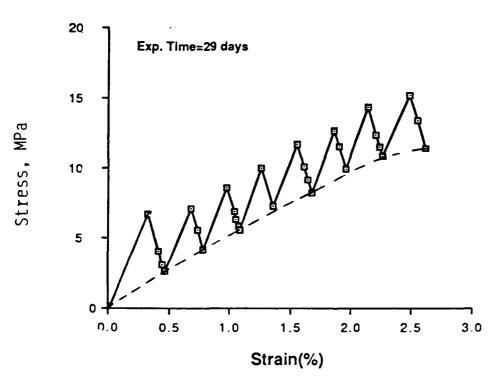


Figure 3. Intermittent stress-strain-relaxation testing of BRD-07A at 120°F/85% RH.